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(54) **A process for drying and expanding microspheres.**

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Description

The present invention relates to a process for drying and expanding polymeric particles. In particular the invention relates to a process for drying and expanding particles which particles comprise a thermoplastic shell encapsulating a blowing agent. The invention is particularly concerned with drying and expanding microspheres.

Expandable microspheres, their production and use are disclosed in the US patent 3,615,972. The thermoplastic shell of these particles can for example consist of polymers or copolymers formed from monomers such as vinyl chloride, vinylidene chloride, acrylonitrile, methacrylate or styrene. The particle size of the unexpanded spheres, and thus also of the expanded spheres, can vary within wide limits and is selected with respect to the desired properties of the finished product. Particle sizes for unexpanded spheres can for example be 1 µm to 1 mm, preferably 2 µm to 0.5 mm and particularly 5 µm to 50 µm. The diameter of the particles increase at expansion by a factor 2 to 5. The unexpanded spheres contain volatile, liquid blowing agents which are vaporized at heating. These blowing agents can for example be freons such as trichlorofluoromethane, hydrocarbons such as n-pentane, i-pentane, neo-pentane, butane, i-butane or other blowing agents conventionally used for this type of microspheres. The blowing agent may suitably make up 5 to 30 per cent by weight of the weight of the microspheres. Expancel® is an example of a suitable, commercially available microsphere product which has a thermoplastic shell of a vinylidene/acrylonitrile copolymer and contains iso-butane as a blowing agent.

The expanded particles can be used in several fields of application, e.g. for insulation purposes, in the graphic industry and in the textile industry.

In certain cases it might be suitable to use unexpanded microspheres which are incorporated in a matrix and when this is heated the microspheres expand in situ. However, in the preparation of syntactical foams it is often customary to incorporate pre-expanded microspheres in the matrix. The type of matrix for the expanded particles depends on the particular composition of expanded particles and matrix which it is desired to prepare, and generally the character of the matrix should be such that it does not chemically affect or physically deform the expanded particles, and such that it allows the particles to remain in the prepared composition.

One process for preparing expanded synthetical microspheres is described in US—A—3,611,583. According to this method an exposed layer of microspheres is deposited on a moving surface. The microspheres are deposited from a dispersion of spheres in a liquid, which is a non-solvent for the expanded microspheres, and which contains a flocculating agent. The exposed layer is then brought into a heated zone and the moving surface is heated from a location which at the same time is remote from the microspheres and adjacent to the surface, to a temperature and for such a time as is sufficient for drying and expanding the microspheres. A stream of gas is contacted with the dried and expanded microspheres and dried, expanded microspheres are removed from the moving surface.

This known method is, however, disadvantageous in several aspects, and particularly it should be mentioned that the obtained expanded microspheres are not free-flowing and that a non-uniform product is easily obtained due to the non-uniform expansion which occurs when already expanded particles surround unexpanded particles and heat insulate these, preventing them from being expanded. It is known to improve heat transfer to hollow particles by dispersing them in hot gases (GB—A—1,079,541, DE—A—2,135,721) but it has not been shown possible to avoid agglomeration when both expansion and drying have to take place. It has now been found that it is possible to eliminate these disadvantages and produce uniformly expanded particles which are free-flowing and dry. The microspheres expanded according to the invention also show a good dispersability in e.g. polyesters.

The method of the invention is characterized in that a dispersion of unexpanded microspheres in an inert liquid is atomized in a stream of hot, inert gas of such an amount and temperature that in a first stage dispersion liquid is vaporized and in a second stage the microspheres are expanded by evaporation of the blowing agent and that dry and free-flowing expanded microspheres are separated from the gas. The inert liquid is preferably water and the inert gas is preferably air. The dry content of the dispersion may vary within wide limits and it has been found suitable that the dispersions have a dry content between 25 and 50 per cent, preferably between 35 and 45 per cent. The temperature of the dispersion is not critical and may vary within wide limits, e.g. between environmental temperature (room temperature) and the temperature at which the dispersion is produced. Neither is the temperature of the introduced air critical relative to the risk of over-expansion of the microspheres as the introduced air during the first stage of the drying/expansion-process according to the invention is used to vaporize the water. The temperature and the amount of the continuously supplied air are selected with respect to the temperature and dry content of the dispersion in such a manner that the temperature of the air, which surrounds the microspheres, in the second stage of the process, i.e. the expansion stage, does not exceed 180°C. The air temperature in the expansion stage should suitably be between 80 and 150°C, and preferably between 90 and 120°C, while the temperature of the supplied air can vary between 140 and 300, preferably between 160 and 250 and particularly between 180 and 200°C. If the temperature exceeds 180°C during the expansion stage the microspheres will be over-expanded, they will collapse and a non-desired product will be obtained. A suitable manner of adjusting the

expansion temperature within the mentioned limits is to adjust the amount of the introduced dispersion with the aid of the pump which feeds the dispersion. The atomization of the dispersion can be carried out in a so-called atomizer or a spraying device which for example can consist of one or more discs or nozzles. To make the expansion as uniform as possible and to avoid clogging of the atomizer the dispersion supplied to the atomizer should be as homogenous as possible and it should thus have been subjected to careful stirring, optionally in combination with screening, immediately before it is fed to the atomizer.

The process of the invention is suitably carried out in an elongated device, which preferably is tubular. In one end of the device an atomizing means for the dispersion of microspheres and water and an inlet for the hot air are arranged. In the other end of the device there is an outlet for air containing expanded microspheres, which outlet is connected to a collecting device for the expanded microspheres, e.g. a filter or a cyclone.

It has been found suitable to carry out the process of the invention in a conventional spray dryer.

Compared with the known method for drying and expanding microspheres, disclosed in the U.S. patent specification No. 3,611,583 mentioned earlier, the process of the present invention gives, besides the earlier mentioned advantages, also the advantage of making it possible to work with comparatively small amounts of dispersion in special-built hot air dryers, while the known method, which is stationary, requires large volumes. Further, at the known method problems easily arise at the supply of the dispersion as it is required to deposit an even and thin layer of the dispersion to a belt, while as has been mentioned above, according to the present invention the dispersion is simply pumped to the atomizer, which divides it finely.

The invention is further illustrated in the following non-limiting example.

Example

A 44-per cent mixture of microspheres in water was prepared. The microspheres had a shell of thermoplastic material of vinylidene chloride and acrylonitrile and the blowing agent was isobutane. To secure a homogenous dispersion the mixture of microspheres and water was carefully stirred and the obtained dispersion was passed through a sieve. The amounts of supplied dispersion and hot air were such that a temperature of about 200°C of the supplied air was suitable to achieve the desired temperature of 100—110°C in the later part of the spray dryer.

During the first part of the process in the spray dryer all heat energy is required to remove the water which surrounds the microspheres. When the microspheres approach the part of the spray dryer where the temperature has dropped to about 100°C the water has been evaporated and the microspheres have dried, and the expansion can now begin.

The microspheres which do not expand (3—15 µm) will be very heavy relative to their volume and they fall down into a cyclone separator while the expanded microspheres continue to a filter where they are collected. The obtained microspheres were free-flowing, they had a dry content of 99% and a density of 30—35 kg/m^3.

Claims

1. A process for drying and expanding heat-expandable microspheres having a thermoplastic shell encapsulating a blowing agent, characterized in, that a dispersion of the unexpanded microspheres in an inert liquid is atomized in a stream of hot, inert gas of such an amount and temperature that in a first stage dispersion liquid is vaporized and in a second stage the microspheres are expanded by evaporation of the blowing agent and that dry and free-flowing expanded microspheres are separated from the gas.

2. A process according to claim 1, characterized in, that the liquid is water and the inert gas is air.

3. A process according to claim 1 or 2, characterized in, that the dispersion of microspheres has a dry content of 25 to 50 per cent.

4. A process according to claim 1, 2 or 3, characterized in, that the temperature in the mixture of microspheres and air at the final stage of expansion is 80—150, preferably 90—120°C.

5. A process according to any of claims 1 to 4, characterized in, that the temperature of the entering air is between 140 and 300, preferably between 160 and 250 and most preferably between 180 and 200°C.

6. A process according to any of claims 1 to 5, characterized in, that the shell of the microspheres consists of a copolymer of vinylidene chloride and acrylonitrile.

7. A process according to any of claims 1 to 6, characterized in, that the blowing agent is isobutane.

8. A process according to any of claims 1 to 7, characterized in, that the atomization is carried out with the aid of atomizing discs or nozzles.

9. A process for drying and expanding microspheres according to any of claims 1 to 8, characterized in, that the process is carried out in a spray dryer.

10. A process according to any of claims 1 to 9, characterized in, that the expanded microspheres are collected in a collecting device which separates air, such as a filter or a cyclone.

Patentansprüche

1. Verfahren zum Trocknen und Ausdehnen oder Aufblähen von hitzeexpandierbaren Mikrokugeln, welche eine thermoplastische Hülle haben, die ein Treibmittel einschließt, dadurch gekennzeichnet, daß eine Dispersion der nicht expandierten Mikrokugeln in einer inerten

Flüssigkeit in einem Strom von heißem inertem Gas einer solchen Menge und Temperatur zerstäubt wird, daß in einer ersten Stufe die Dispersionsflüssigkeit verdampft und in einer zweiten Stufe die Mikrokugeln durch Verdampfung des Treibmittels aufgebläht werden und daß trokkene und frei fließende aufgeblähte Mikrokugeln von dem Gas abgetrennt werden.

2. Verfahren gemäß Anspruch 1, dadurch gekennzeichnet, daß die Flüssigkeit Wasser ist und das inerte Gas Luft ist.

3. Verfahren gemäß Anspruch 1 oder 2, dadurch gekennzeichnet, daß die Dispersion der Mikrokugeln einen Trockengehalt von 25 bis 50 % hat.

4. Verfahren gemäß Anspruch 1, 2 oder 3, dadurch gekennzeichnet, daß die Temperatur in dem Gemisch der Mikrokugeln und Luft bei der Endstufe der Ausdehnung 81—150, vorzugsweise 90—120°C ist.

5. Verfahren gemäß einem der Ansprüche 1 bis 4, dadurch gekennzeichnet, daß die Temperatur der eintretenden Luft zwischen 140 und 300, vorzugsweise zwischen 160 und 250 und ganz vorzugsweise zwischen 180 und 200°C ist.

6. Verfahren gemäß einem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß die Hülle der Mikrokugeln aus einem Copolymeren von Vinylidenchlorid und Acrylnitril besteht.

7. Verfahren gemäß einem der Ansprüche 1 bis 6, dadurch gekennzeichnet, daß das Treibmittel bzw. Aufblähmittel iso-Butan ist.

8. Verfahren gemäß einem der Ansprüche 1 bis 7, dadurch gekennzeichnet, daß die Zerstäubung mit Hilfe von Zerstäubungsscheiben oder -düsen durchgeführt wird.

9. Verfahren zum Trocknen und Aufblähen von Mikrokugeln gemäß einem der Ansprüche 1 bis 8, dadurch gekennzeichnet, daß das Verfahren in einem Sprühtrockner durchgeführt wird.

10. Verfahren gemäß einem der Ansprüche 1 bis 9, dadurch gekennzeichnet, daß die aufgeblähten Mikrokugeln in einer Sammelvorrichtung gesammelt werden, welche Luft abtrennt, wie ein Filter oder ein Zyklon.

Revendications

1. Procédé de séchage et d'expansion de microsphères expansibles comprenant une capsule thermoplastique enrobant un agent gonflant, caractérisé en ce qu'une dispersion de microsphères non expansées dans un liquide inerte est atomisée dans un courant de gaz chaud inerte en une quantité et à une température telles que dans un premier stade le liquide dispersant soit vaporisé et que dans un second stade les microsphères soient expansées par évaporation de l'agent gonflant et en ce que les microsphères expansées sèches et s'écoulant librement sont séparées du gaz.

2. Procédé selon la revendication 1, caractérisé en ce que le liquide est de l'eau et le gaz inerte est de l'air.

3. Procédé selon la revendication 1 ou 2, caractérisé en ce que la dispersion des microsphères présente une teneur en matière sèche entre 25 et 50 %.

4. Procédé selon la revendication 1, 2 ou 3, caractérisé en ce que la température du mélange de microsphères et d'air dans le stade final de l'expansion est comprise entre 80 et 150°C, de préférence entre 90 et 120°C.

5. Procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce que la température de l'air au moment de son introduction est comprise entre 140 et 300°C, plus avantageusement entre 160 et 250°C et de préférence entre 180 et 200°C.

6. Procédé selon l'une quelconque des revendications 1 à 5, caractérisé en ce que la capsule des microsphères consiste en un copolymère de chlorure de vinylidène et d'acrylonitrile.

7. Procédé selon l'une quelconque des revendications 1 à 6, caractérisé en ce que l'agent gonflant est l'isobutane.

8. Procédé selon l'une quelconque des revendications 1 à 7, caractérisé en ce que l'atomisation est effectuée à l'aide de disques ou de tuyères d'atomisation.

9. Procédé de séchage et d'expansion de microsphères selon l'une quelconque des revendications 1 à 8, caractérisé en ce que le procédé est effectué dans un sécheur à pulvérisation.

10. Procédé selon l'une quelconque des revendications 1 à 9, caractérisé en ce que les microsphères expansées sont recueillies dans un dispositif récepteur qui élimine l'air, tel qu'un filtre ou un séparateur à cyclone.